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**SUPERPLASTICITY OF ULTRA-FINE GRAINED ALUMINIUM ALLOYS**

**Final Technical Report**

by

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## LIST OF ALL PUBLICATIONS

1. Stolyarov, V.V. Latysh, V.A. Shundalov, D.A. Salimonenko, R.K. Islamgaliev, R.Z. Valiev. Influence of severe plastic deformation on aging effect of Al-Zn-Cu-Zr alloy. Mater. Sci & Eng. A234-236 (1997) pp.339-342.
2. P.B. Berbon et al., Optimizing the Processing of a Commercial Al-Based Alloy for High Strain Rate Superplasticity, Proc. The Int. Conf. On superplasticity, Mie, Japan, August 1997, (to be published).
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4. R.Z.Valiev and R.K.Islamgaliev. Microstructural aspects of superplasticity in ultrafine-grained alloys. TMS Annual Meeting. Superplasticity and Superplastic Forming, San Antonio, Texas, February 14-19, 1998 (to be published).
5. P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon, "High strain rate superplasticity in a commercial cast alloy", Nature, (submitted).
6. P.B. Berbon, N.K. Tsenev, R.Z. Valiev, M.Furukawa, Z. Horita, M. Nemoto and T.G. Langdon, "High strain rate superplasticity in fine-grained commercial Al alloys processed by equal-channel angular pressing", Proc. the NATO ASI on Advanced Al-alloys. Poland, September 1997 (to be published).
7. P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto, N.K. Tsenev, R.Z. Valiev and T.G. Langdon. Processing of aluminium alloys for high strain rate superplasticity. Proc. The TMS Fall meeting, 1998 (to be published).

## Contents

	page
Abstract	1
Introduction	1
Materials and experimental procedures	2
Processing of UFG Al based alloys	3
Structural characterization and mechanical behaviour of UFG Al-based alloys (results and discussion)	5
Conclusions	11
Recommendations	11
Reference	11

## ABSTRACT

Superplasticity requires a fine grain size, typically in the range of 1 - 10  $\mu\text{m}$ . Experiments have established that much finer grain sizes, in the near-nanometer range, may be achieved in Al-based alloys by using an intense plastic straining technique such as equal-channel angular (ECA) pressing. This report presents the microstructural characteristics and mechanical properties of several ultrafine-grained Al-based alloys processed by ECA pressing. The first demonstration of enhanced superplasticity at low temperatures and high strain rates revealed in commercial Al-alloys, having ultrafine-grained structure, are described.

**Keywords:** *ultrafine-grained materials, aluminium alloys, equal-channel angular pressing, superplasticity, grain boundaries.*

## INTRODUCTION

Ultrafine-grained (UFG) materials (nano-and submicrocrystalline) having a grain size of tens and hundreds nanometers recently has aroused interest among researchers in the material science field [1, 2]. In respect to their mechanical properties one should note a hope to obtain extremely strength states that can be expected in the case of strong grain refinement according to the Hall-Petch relationship. Another important aspect is superplasticity in these materials. Superplastic forming has now become established as an important industrial tool for the fabrication of complex shapes from sheet metals. However, optimum superplastic flow is usually attained at strain rates of the order of  $10^{-4}$  -  $10^{-3}$   $\text{s}^{-1}$ , and these rates are generally too low for the successful utilization of superplastic forming in situations where it is necessary to fabricate many thousands of identical components [4]. Following the existing models one can expect that superplasticity can be achieved at relatively low temperatures and/or high strain rates by reducing grain size of materials into a submicrometer or nanometer range [3-5]. However, until recent times, there were problems with such experimental studies, since UFG materials processed by traditional techniques, namely, gas condensation [1, 2] and ball milling [6] followed by consolidation, are characterized by some residual porosity and it is rather difficult to fabricate large samples for subsequent tensile test. Nevertheless, as it was demonstrated recently in our laboratory [7, 8], the mentioned problems can be overcome by processing UFG structure through a procedure of severe plastic deformation, i.e. very high plastic strain performed at relatively low temperature (usually less than  $0.4 T_m$ ) under high imposed pressure. Although the fact of significant structure refinement under heavy deformation, in particular by drawing and rolling, has long been well known [9, 10], but only recently it has been shown that in contrast to previous works demonstrating a formation of cell structures during large plastic straining, intense plastic deformation under high applied pressure can provide formation of ultrafine grained structures with high angle grain boundaries [8,11]. This method named severe plastic deformation (SPD) was realized via special modes of mechanical deformation such as torsion straining (TS) under high pressure, equal channel angular (ECA) pressing and others. It is already established that intense straining is capable of producing materials which exhibit superplasticity at relatively low testing temperatures [12,13] and there has been the demonstration of the potential for realizing high strain rate superplasticity (HSR SP) [13]. At the same time, it should be noticed that the microstructure of metals processed by severe plastic deformation is characterized not only by a very small grain size with high angle misorientation of neighboring grains, but also a specific defect structure of grain boundaries, morphology of second phases and crystallographic texture, this all is important for achieving enhanced superplasticity. This indicates an attractive vista of investigations of superplasticity in UFG alloys. Two main tasks of a special interest here: first, development of a ECA pressing technique for processing bulk samples with homogeneous UFG structure and high angle grain boundaries and, second, demonstration of enhanced superplasticity in UFG materials. These tasks are in the focus of the present Project. The presented Final Report describes the results of carrying out of the project «Superplasticity of the ultra-fine grained Al alloys» ARO № 68171-97-C-9006, based on a mutual cooperation of teams of Prof. R.Z. Valiev (IPAM USATU, Ufa) and T. Langdon (USC, Los Angeles). The scientific team of Professor R.Z. Valiev has been responsible for processing, some structural characterization and investigations of mechanical properties of the processed Al-based alloys. The Professor Langdon's team has been responsible for detailed investigations of mechanical behaviour of UFG alloys and also for some structural studies.

## MATERIALS AND EXPERIMENTAL PROCEDURES

Two recently developed techniques of severe plastic deformation are illustrated in Fig.1. Equal-channel-angular (ECA) pressing, shown in Fig.1(a) is a procedure in which the sample is pressed under a load  $P$  through two channels of equal cross-section intersecting at an angle  $\Phi$  and the pressing are repeated to attain the required level of strain. This procedure was developed many years ago in order to introduce an intense plastic strain into materials with no change in the cross-sectional area [14] and it is now established as a method for attaining a submicrometer or nanometer grain size [7, 8]. The ECA pressing was conducted using the facility illustrated schematically in Fig.2. Two channels, equal in cross-section, intersect within a die. The test sample is machined to fit tightly within the die and it is then pressed through the die using a plunger. The strain imposed on the sample in a single passage through the die is determined by the values of the two angles,  $\Phi$  and  $\Psi$ , shown in Fig. 2. Since the cross-section of the sample is unchanged by passage through the die, additional pressings may be conducted on the same sample to attain a high strain. It has been shown teoretically [15, 16], and confirmed experimentally [17], that the strain,  $\epsilon_N$ , associated with a total of  $N$  pressings through the die may be expressed as

$$\epsilon_N = \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) + \Psi \operatorname{cosec} \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) \right] \quad (1)$$

In the present experiments, the die was constructed with  $\Phi = 90^\circ$  and  $\Psi = 0^\circ$  so that, from eq. (1), a strain of  $\sim 1$  is introduced in each passage through the die. A total equivalent true plastic strain has been up of order of 12. The produced samples were in a form of rod, 20 mm in diameter and  $60 \div 80$  mm in length.

Torsion straining under high pressure shown in Fig.1 (b) is a procedure in which disk samples are subjected to high plastic deformation by torsion straining under an imposed pressure. Torsion straining was carried out at room temperature to a true logarithmic strain of 7 and under a pressure of 5 GPa. The samples processed by this technique were in a form of disks with a diameter of 12 mm or 20 mm and a thickness of 0.2 mm or 1 mm.

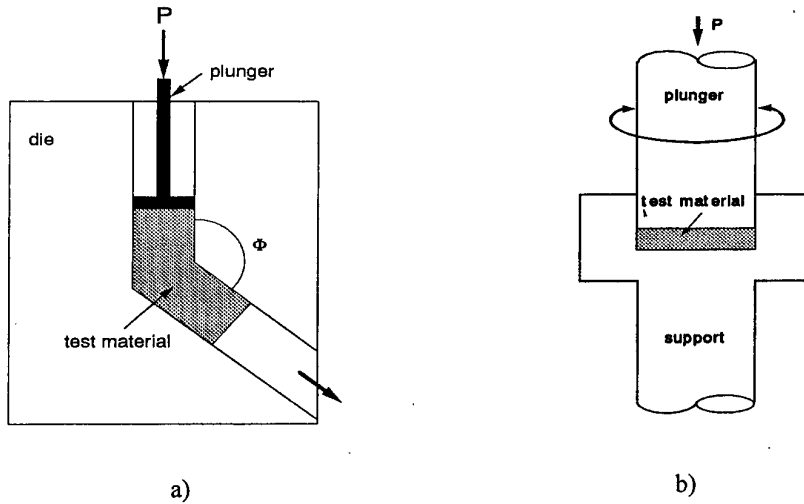


Fig. 1. Principles of equal-channel angular pressing (a) and torsion straining (b).

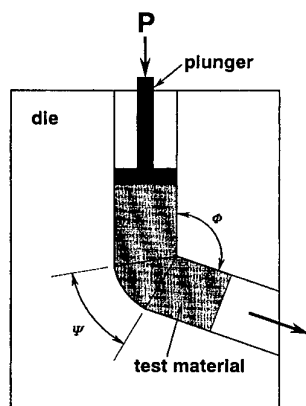


Fig. 2. Schematic representation of a die for ECA pressing.

The experimental materials were the commercial alloy Supral with the chemical composition, Al-6%Cu-0.5%Zr produced in Great Britain, the commercial aluminium alloys 1420 (Al-5%Mg-2%Li-0.1%Zr), 1421 (Al-5%Mg-2%Li-0.1%Zr-0.2%Sc), 1460 (Al-2.5%Cu-2%Li-0.1%Zr) fabricated in Russia and model alloy Al-4%Cu-0.5%Zr. The commercial alloy Supral is widely used in industry for superplastic forming. The maximum elongation ~1000% and the low flow stress about 10 Mpa are usually observed in this alloy with the grain size about 10 micrometers at the temperature 450°C and the strain rate  $10^{-3} \text{ s}^{-1}$  [4]. An increase of superplastic properties due to formation of UFG structure is an actual task here. As compared to traditional aluminium alloys the commercial alloys with Li additions are characterized by reduced 10% specific weight and elevated 4-5% elastic modulus [18]. Due to the mentioned properties these materials are rather advanced for aerospace and motor-car industries. In this connection, the investigation of enhanced superplasticity in the given alloys is of significant interest.

The structure of samples was examined by transmission electron microscopy. Disks for thin foils were cut out by the spark technique and thinned by double-jet electrolytic polishing. The examination of the microstructure was performed by the methods of dark and bright field images, and by microdiffraction. Electron diffraction patterns were taken from an area of about one  $\mu\text{m}^2$ . A mean grain size was determined as a mean value of a grain size diameter by averaging more than 100 grains. Mechanical tests of superplastic samples were conducted by pulling tensile specimens to failure in air over a range of temperatures at initial strain rates from  $\sim 10^{-5}$  to  $10^{-1} \text{ s}^{-1}$ . Further details on processing and tests are given elsewhere (see the list of submitted papers).

In the as-received state all three alloys with Li additions were hot rolled and aged plates. All these alloys are characterized by the presence of  $\delta$ - (AlLi) and  $\delta'$ - ( $\text{Al}_3\text{Li}$ ) phases. The superstructural spots observed on electron diffraction patterns [19] confirm presence of these phases. In addition to the  $\delta$ - and  $\delta'$ - phase particles each alloy has its own disperse precipitates determined by chemical composition of an alloy. Thus, in 1420 and 1421 alloys the cubic ( $a=2.02 \text{ nm}$ )  $\text{S}(\text{Al}_2\text{MgLi})$  phase with the orientation ratio  $(\bar{1}10)_\text{S}/(\bar{1}10)_{\text{Al}}$ ;  $[110]_\text{S}/[111]_{\text{Al}}$  is revealed [19]. At the same time particles of the  $\gamma(\text{Mg}_{17}\text{Al}_{12})$  phase of an a-Mn type with a lattice parameter of  $a = 1.052 \text{ nm}$  are also observed in these alloys [18]. In addition to the considered precipitates some disperse precipitates of  $\text{Al}_3\text{Sc}$  are present in the 1421 alloy and, as a rule, precipitates of the  $\delta'(\text{Al}_3\text{Li})$ - phase are observed on them at low temperature ageing.

## PROCESSING OF UFG AL-BASED ALLOYS

The most interesting technique in the field of superplasticity of UFG alloys is ECA pressing because a very high strain can be introduced into samples having large sizes. There are several processing parameters, which can be changed during a fabrication ultrafine-grained structures by severe plastic deformation, namely, temperature and strain rate, imposed pressure, amount of straining, lubrication and others. These parameters were controlled in order to process a homogeneous structures and grain size as small as possible.

With this aim we provide the optimization of the temperature of ECA pressing as well as the optimization of a route of sample movement at following passes. Three investigated routes A, B, C are shown in Fig. 3. These routes are distinguished by a rotation angle of samples and, consequently, acting slip systems. Structural investigations showed that ECA pressing after 8 and more passes in the temperature interval  $T = 200$ -

400°C can strongly refine microstructure using all three routes, but only strain by regime B provided successful formation of homogeneous ultra-fine grained structure in investigated alloys, as it considered below. In other cases many elongated grains and subgrains were observed in microstructure.

Additional experiments made focus special attention on tribological conditions of the process. The application of a special lubricant on a graphite base in the initial stage of investigations resulted in tooling damage due to low efficiency coating of a sample surface. This resulted in formation of coarsening at the instrument's surface and sticking to forming parts of the die-set. Three new compositions of the lubricant on a graphite base and electrocopper plating of samples in combination with lubricants were tested to solve this task. New lubricants on graphite base were distinguished by brands of graphite powder and binders. In the temperature range of 200-400°C the lubricant on scaly type graphite base demonstrated the best operation properties. As a result, strain efforts were decreased and no cases of adhesion were observed. Copper coating in combination with the optimal lubricant positively influences the reliability of the process but increases its labor-intensity.

One more way of increasing efficiency of ECA pressing is an application of a special die-set with the active friction (Fig.3). The given design allowed us not only to decrease a load by 1/3 and to increase a surface quality but also provided an opportunity to increase a degree of material processing via stable fixing of pressing sample movement that consequently contributed to obtaining of homogeneous UFG structure within the whole sample volume for a less number of pressing cycles.

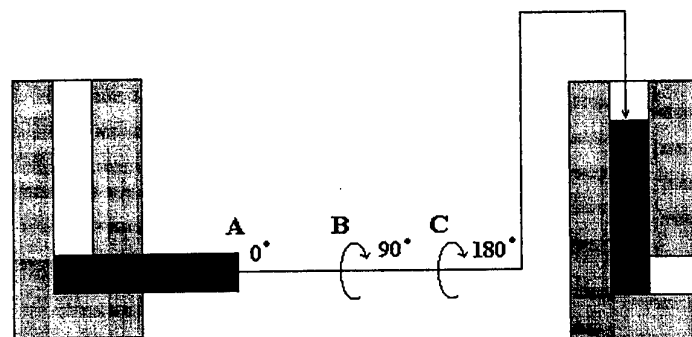


Fig. 3. The routes of samples during ECA pressing.

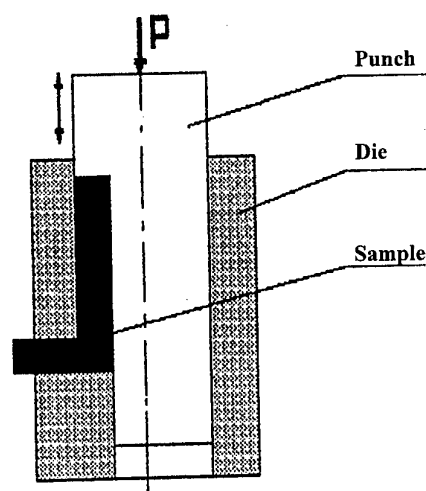


Fig. 4. Scheme of the die- set with active friction forces.



## STRUCTURAL CHARACTERIZATION AND MECHANICAL BEHAVIOUR OF UFG AL-BASED ALLOYS (RESULTS AND DISCUSSION).

As noted above, there are two main advantages of superplasticity application in UFG materials: firstly, possibility of a essentially decrease of temperature of superplasticity and, secondly, realization of superplasticity at significantly higher strain rates. Both of effects directly depend on structure of fabricated materials, and below, we shall consider main structural characteristics of Al alloys subjected to ECA pressing. Then the results of investigations of mechanical properties of these materials illustrating low and high strain rate superplasticity and demonstrating an opportunity to achieve a high strength state in UFG Al alloys will be shown.

### Low temperature superplasticity

Figure 5 shows the typical microstructure of the Al-4%Cu-0.5%Zr alloy after ECA pressing, together with selected area electron diffraction (SAED) pattern obtained from region with a diameter of  $\sim 1\mu\text{m}$ . Inspection showed that the microstructure is enough homogeneous and a mean grain size is about 150nm. This is the microstructure of granular type, i.e. having high angle grain boundaries because the diffraction pattern presents numerous spots arranged in circles. Such diffraction pattern provides the evidence for high angle misorientations being present in the structure and this is typical for different UFG materials processed by severe plastic deformation [7, 8]. Recently, the presence of high angle grain boundaries in UFG materials processed by severe plastic deformation was directly confirmed by direct measurement of misorientation of separate grains [20]. As it is seen on Fig. 5 in the structure, some grain boundaries are visible and these appear to be mostly curved or wavy. There are also grain boundaries that are poorly defined. The contrast within the grains is not uniform but often changes in complex way, that indicates high level internal stresses and elastic distortions of crystal lattice. All of these observations suggest that the grain boundaries are in a non-equilibrium state [7].



Fig. 5. TEM micrograph of the Al-4%Cu-0.5%Zr alloy after ECA pressing.

Let us consider now results of mechanical tests. Specimens of ultrafine grained alloys produced by ECA pressing were pulled in tension at constant displacement rates at different temperatures. Figure 6 shows the view of specimens of the Al-4% Cu-0,5% Zr alloy tensiled at a temperature of 523 K with various values of an initial strain rate from  $1.4 \times 10^{-5}$  to  $1.4 \times 10^{-2} \text{ s}^{-1}$ . It is apparent from the results that the alloy with an ultrafine grain size of 150 nm is capable of elongation was  $\sim 850\%$  at an initial strain rate of  $1.4 \times 10^{-4} \text{ s}^{-1}$ . The strain rate sensitivity,  $m$ , for this case was equal 0,46. By contrast, the same alloy with a grain size of  $8\mu\text{m}$  display the similar superplastic behavior at a temperature of 773 K only [21]. These results have demonstrated superplasticity at low temperatures in Al-Cu-Zr alloy after ECA pressing. The nature of this unusual phenomenon has recently been discussed in our work [13].

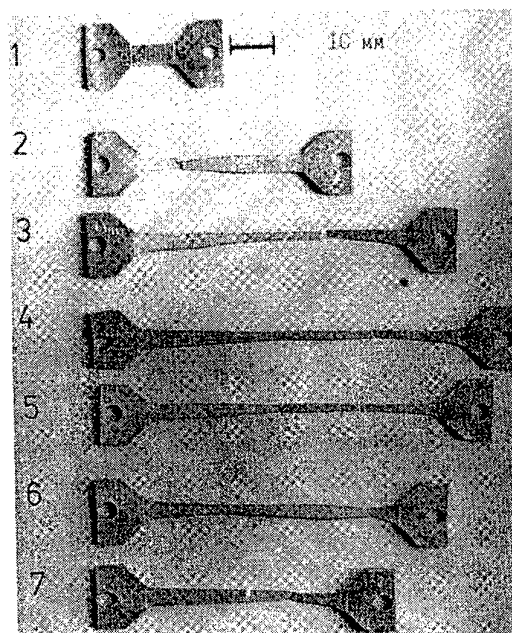


Fig. 6. Specimens of the Al-4%Cu-0.5%Zr alloy prepared by ECA-pressing and pulled in tension at a temperature 523 K with different strain rates:  
 (1) a starting specimen, (2)  $2.8 \times 10^{-5} \text{ s}^{-1}$ , (3)  $7.0 \times 10^{-5} \text{ s}^{-1}$ , (4)  $1.4 \times 10^{-4} \text{ s}^{-1}$ ,  
 (5)  $2.8 \times 10^{-4} \text{ s}^{-1}$ , (6)  $7.0 \times 10^{-4} \text{ s}^{-1}$ , (7)  $1.4 \times 10^{-3} \text{ s}^{-1}$ .

#### High strain rate superplasticity.

Recent discovery and investigations of high strain rate superplasticity (HSRS), i.e. superplastic behaviour at strain rates above  $10^{-2} \text{ s}^{-1}$  have attracted much interest among researchers [4]. This is attributed to understanding of mechanisms of this unusual phenomenon and tempting advantages of very promising its practical application, since a strain rate increase provides a significant reduction in time of parts forming from superplastic alloys. However, up to now HSRS has been observed in a number of the so-called advanced metallic alloys including metallic matrix compounds, mechanically alloyed materials, alloys produced out of powders. In this connection, the discovery of HSRS in conventional commercial alloys may have a special practical importance. Following the well-known dependence that a rate of superplastic deformation increases with the decreasing grain size one can expect the display of HSRS in alloys having a nano- and submicron grain size [4, 5, 13]. However, as it was discussed a head, up to recent time there have been difficulties connected with processing of such ultra fine-grained structures in bulk samples and which were overcome due to the development of the method of severe plastic deformation. This part of the Report deals with the first demonstration of HSRS in commercial Al-alloys, subjected to ECA pressing.

In this Report four commercial alloys were used for the investigations of superplastic behaviour at high strain rates: Supral (Al-6%Cu-0.5%Zr), 1420 (Al-5.5%Mg-2.1%Li-0.12%Zr), 1460 (Al-2.65%Cu-2.1%Li-0.12%Zr-0.07%Sc-0.25%Si) and 1421 (Al-5%Mg-2.2%Li-0.12%Zr-0.2%Sc).

Fig. 7 shows a typical microstructure of the 1420 alloy and the associated selected area diffraction pattern. Examination of Fig. 7. reveals an essentially homogenous UFG microstructure consisting of reasonably equiaxed grains, separated by high angle boundaries with an average grain size which was measured as  $1.2 \mu\text{m}$ . The selected area diffraction pattern exhibits circles showing the presence of high angle grain boundaries. It should be noticed that ultrafine grains introduced into the Al-Ag-Li-Zr alloy used in the present investigation display remarkable stability such that significant grain growth occurs only at temperatures above  $\sim 650 \text{ K}$ , corresponding to  $\sim 0.6 T_m$  where  $T_m$  is the absolute melting temperature of the material. This stability is attributed to the presence of  $\beta'$  -  $\text{Al}_3\text{Zr}$  precipitates in this alloy because these precipitates will be stable at these high temperatures. Since superplasticity is a diffusion-controlled process, the results suggest that the Al-Mg-Li-Zr alloy may be a suitable candidate material for the development of superplasticity at temperatures in the range of  $\sim 550 - 700 \text{ K}$ : these

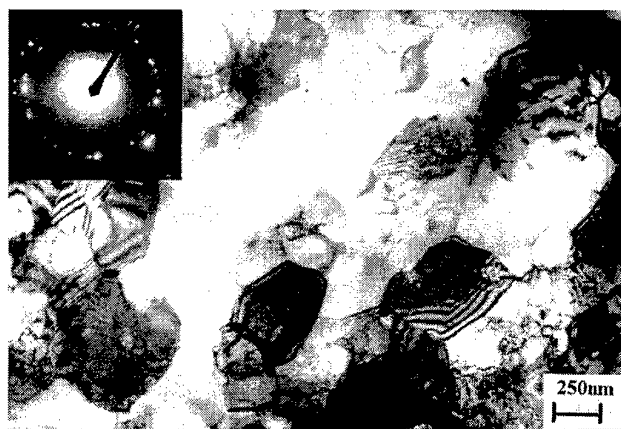


Fig. 7. Microstructure and diffraction pattern in the 1420 alloy after ECA pressing.

temperatures are attractive because they avoid problems associated with the depletion of Li and Mg at higher temperatures.

Let us consider the results of mechanical testing. Figure 8 shows a plot of stress,  $\sigma$ , versus strain,  $\epsilon$ , for three samples pulled at initial strain rates,  $\dot{\epsilon}$ , from  $1 \times 10^{-2}$  to  $1 \text{ s}^{-1}$ . Each of these curves shows an initial period of strain hardening which is typical of ECA-pressed materials where there is some relaxation of the internal stresses, and this is followed by a subsequent period of weakening except for the sample tested at  $1 \times 10^{-2} \text{ s}^{-1}$  where the test discontinued prior to failure. Thus, whereas the unpressed material exhibits only modest elongations even at relatively low strain rates, the ECA-pressed alloy gives elongations as high as 1180% without failure at  $1 \times 10^{-2} \text{ s}^{-1}$  and 910% at  $1 \times 10^{-1} \text{ s}^{-1}$ . The appearance of these two specimens after testing is shown in Fig. 9 where the upper sample is in untested condition. These specimens represent a very clear demonstration of HSR SP, especially when it is noted that the high elongation of  $> 1000\%$  is achieved at  $1 \times 10^{-2} \text{ s}^{-1}$  without the development of any visible necking within the gauge length.

One more material used for investigations of superplastic properties was Supral. Following ECA pressing, the measured grain size in Supral processed by ECA pressing was  $\sim 0.5 \mu\text{m}$  (Fig. 10). An example of the true stress-strain curves is shown in Fig. 11 for two tests conducted at 573 K and a single test at 623 K. It is apparent that these specimens also exhibit very high tensile ductility, with elongations up to a maximum of 970% at 573 K when testing with a strain rate of  $1 \times 10^{-2} \text{ s}^{-1}$ . The appearance of these three specimens after testing is shown in Fig. 12. These results are significant because they demonstrate not only the potential for HSR SP but also that very high ductilities may be achieved at relatively low testing temperatures. For example, a previous investigation of the Supral 100 alloy revealed elongations of up to  $> 1000\%$  but these occurred at the much higher testing temperatures of  $\sim 750 \text{ K}$  and at the low strain rates  $\sim 10^{-4}$  to  $10^{-3} \text{ s}^{-1}$  [4].

The appearance of three tensile specimens tested to failure at strain rates of  $1 \times 10^{-2}$  and  $1 \times 10^{-1} \text{ s}^{-1}$  is shown in Fig. 12. These specimens also exhibit HSR SP, with a maximum elongation of 970% recorded with a strain rate of  $1 \times 10^{-2} \text{ s}^{-1}$  at a testing temperature of 573 K. The high elongation of 740% achieved at a strain of  $1 \times 10^{-1} \text{ s}^{-1}$  at 623 K suggests that even greater elongations may be achieved at this higher temperature but a more detailed investigation is required to fully examine this possibility.

Very high elongations were revealed also during tensile tests of 1460 and 1421 alloys, subjected to ECA pressing. For example, Fig. 13 shows the appearance of tensile specimens of the 1421 alloy after ECA pressing, which illustrates HSR superplasticity at 623 K.

The results described on this report represent the first demonstration of high strain rate superplasticity in conventional commercial Al alloys and they were achieved by introducing ultrafine grain sizes into the materials by intense plastic straining through equal-channel angular pressing. For each alloy, the strain introduced by ECA pressing was very high ( $\sim 12$ ); as demonstrated in our earlier report, significantly less tensile ductility, and no evidence for HSR SP, was obtained in experiments on the Al-Mg-Li-Zr alloy when the total strain introduced by ECA pressing was only  $\sim 4$ .

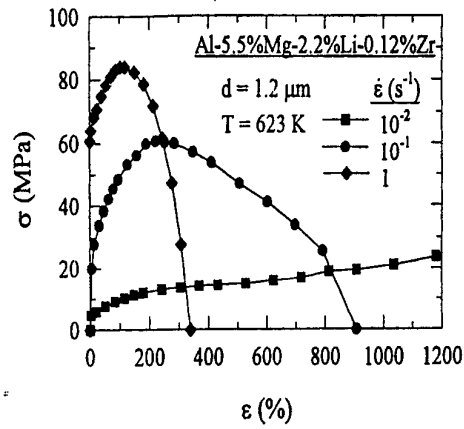


Fig.8. Stress versus strain curves of the ECA pressed Al-Mg-Li-Zr alloy.

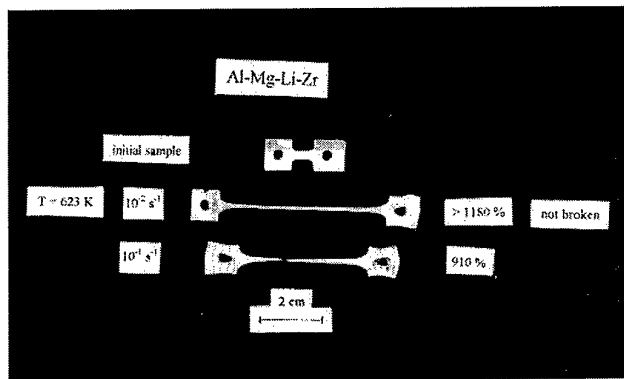


Fig.9. Appearance of tensile specimens of the Al-Mg-Li-Zr alloy, prepared by ECA pressing, which demonstrates HSR SP.

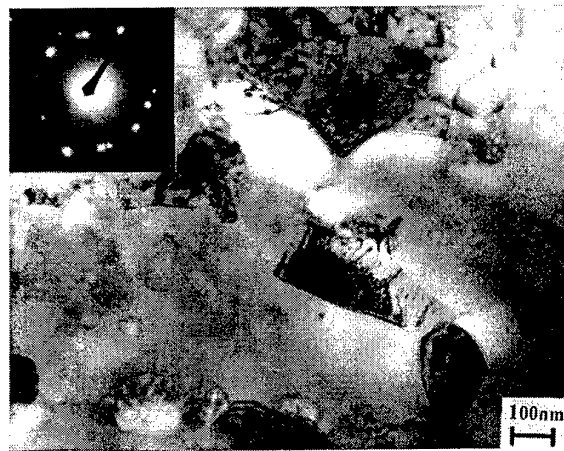


Fig. 10. Microstructure and diffraction pattern in the Supral after ECA pressing.

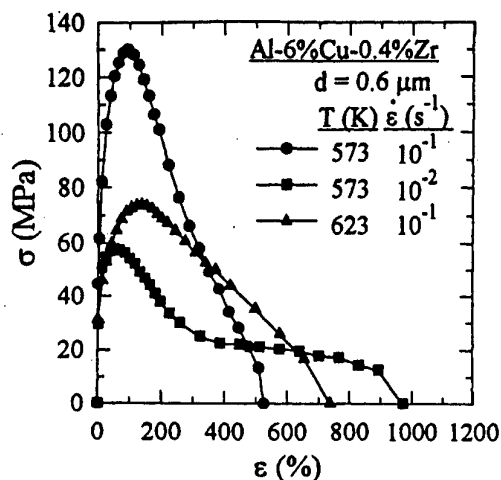


Fig. 11. Stress versus strain curved of the ECA pressed Supral alloy.

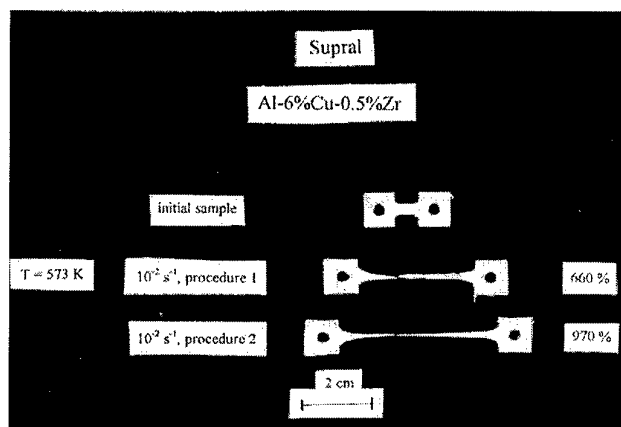


Fig.12. A demonstration of HSR SP in the commercial Supral alloy

A comparison with data tabulated in an extensive review of HSR SP materials [22] shows that the present results represent not only the first evidence of HSR SP achieved through the use of ECA pressing but, in addition, the elongations attained in the present experiments are exceptionally high even by comparison with normal HSR SP materials where the elongations to failure are often in the range of ~500-800%. For example, for Al alloys fabricated using powder metallurgy techniques, the highest tabulated elongation is 1060% for an Al-Zn-Mg-Cu-Zr alloy having a grain size of  $1.2\mu\text{m}$  tested at 788 K with an imposed strain rate of  $7 \times 10^{-2}\text{s}^{-1}$ . This maximum elongation to failure is slightly lower than the elongation of 1180% achieved without failure in the present limited set of experiments on the commercial Al-Mg-Li-Zr alloy.

The present results show also that the increase in strain rate for HSR SP is associated with a significant decrease in the testing temperature. For example, the Al-Cu-Zr alloy typically exhibits optimum superplasticity with elongations pf  $> 1000\%$  at temperatures in the vicinity of  $\sim 700\text{-}750\text{ K}$  and at strain rates of  $\sim 10^{-4}$  to  $10^{-3}\text{s}^{-1}$  [4]. In the present experiments, superplastic elongations were achieved at a temperature of 573 K which is both below the temperatures associated with superplastic flow in the standart recrystallized condition and also reduction in temperature is an especially attractive feature of HSR SP because the lower testing temperature assists in retaining the ultrafine grain size introduced by ECA pressing.

Finally, the present results provide some insight into the possible mechanism associated with HSR SP. In metal matrix composites exhibiting HSR SP, the experiments are often performed at temperatures close to, or even slightly higher than, the solidus temperature for the metal matrix. This has led to the development of a rheological model in which HSR SP takes place in a semi-solid material behaving in a manner similar to a non-

Newtonian fluid [23]. However, theories of this type cannot explain the present results where HSR SP was achieved in two conventional commercial alloys at relatively low testing temperature where there is no local melting at the grain boundaries. The present results therefore serve to demonstrate that the presence of a liquid phase is not a requirement in order to achieve HSR SP in Al-based alloys.

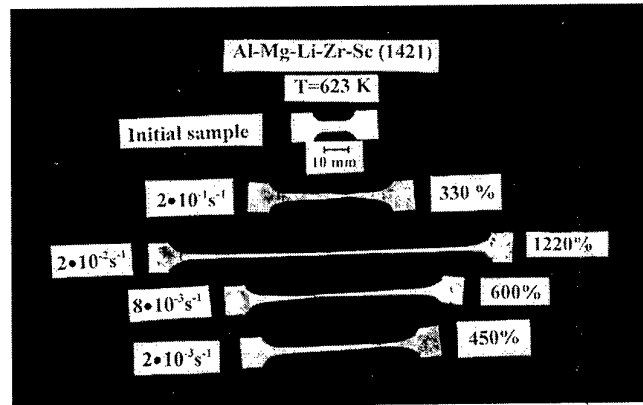


Fig. 13. Appearance of tensile specimens of the 1421 alloy, prepared by ECA pressing, which demonstrates HSR SP.

#### High strength state

Superplasticity in UFG alloys observed at low temperatures or/and high strain rates is also interesting due to the fact that no grain growth happens here. In this connection, the investigation of high strength state achieved in UFG alloys is very actual, because, this state is important for providing high service properties of articles fabricated by superplastic forming. That is why in this project we also carry out the investigations of microhardness of the 1420 alloy subjected to quenching with subsequent torsion straining and ageing [24].

The structural investigations showed that second phase precipitates are absent in the aluminium 1420 alloy subjected to quenching. A mean grain size of an aluminium base matrix was equal to 10  $\mu\text{m}$ . A value of microhardness of the quenched sample was 540 MPa.

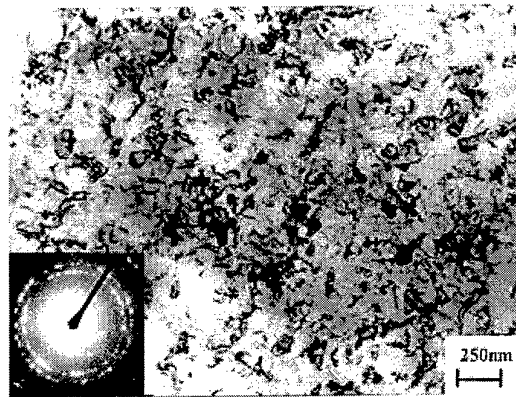


Fig. 14. Typical microstructures of Al alloy 1420 after torsion straining.

Severe plastic deformation by torsion straining of the 1420 alloy led to formation of nanocrystalline structure with a mean grain size of 70 nm (Fig. 14). The TEM studies have revealed the presence of bend extinction contours inside of grains and the occurrence of a specific diffraction contrast on their boundaries testifying a high level of internal elastic stresses. A number of spots arranged in a circle were observed on the diffraction pattern and it testifies to high angle misorientations of neighbouring grains in the structure [11, 13].

After severe torsion straining a significant growth of microhardness up to 1750 MPa was observed, that remained till an annealing temperature of 150°C. A drop of microhardness attributed to structure relaxation and grain growth onset was observed with subsequent annealing temperature. In this connection, ageing of the samples was conducted at a temperature of 120°C at a constant mean grain size. After 120°C temperature ageing a value of microhardness of the 1420 alloy increased to 2300 MPa. The TEM studies did not reveal a noticeable grain growth in the aged alloy but at the same time second phase particles, about 20 nm in size were observed [24]. Al alloys 1421 and 1460 processed by quenching with subsequent torsion straining and ageing demonstrated similar nanostructure, but the level of microhardness exhibits of 2500 MPa and 2800 MPa respectively.

## CONCLUSIONS

1. The developed method of ECA pressing allowed us to fabricate successfully different ultrafine-grained Al-alloys with uniform microstructure having mainly high angle grain boundaries.
2. Unusual, but very attractive phenomena of low temperature and high strain rate superplasticity have been demonstrated in the fabricated ultrafine-grained alloys. The results show that contrary to some implications from experiments on metal matrix composites, partial melting is not a requirement for HSR SP.
3. The formation of ultrafine-grained structures by severe plastic deformation in the Al-alloys leads to ability to reach high strength properties that it is attributed to both the presence of ultrafine grains and dispersive particles.

## RECOMMENDATIONS

Demonstration of low temperature and high strain rate superplasticity in ultrafine-grained alloys opens up a new trend in up-to-date investigations of superplasticity. The study of mechanisms and physical nature of these new kinds of superplastic behaviour is rather important. Here one should attach significance to investigate a structure evolution during superplastic deformation, definition of morphology and a role of disperse particles in enhanced superplasticity of UFG alloys. Development of ECA pressing aiming to obtain even finer structure (up to 50 nm - 100 nm) and to increase non-equilibrium state of grain boundaries, the latter can be achieved by decreasing the temperature of ECA pressing, have considerably high actuality nowadays.

There is a scientific interest in the development of this investigations to extend a range of materials under research including advanced Al composites, which through a smaller grain size and higher thermostability can display enhanced low temperature and high strain rate superplasticity. It also is important to modernize a die and optimize a route for ECA pressing in order to fabricate samples with dimensions of about 50 mm in diameter and 150 mm in length. It would allow to use these large samples to demonstrate a possible utilization of superplasticity UFG Al alloys for production of light and high strengthened articles of a complex shape. Another attractive area of utilization is welding under pressure of UFG Al alloys at superplastic conditions.

Thus taking into account the significant importance of these objectives, it is desirable and preferable to have these investigations as a continuation of the present project.

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